

TRANSMITTAL LETTER

In re Application of:
PERSSON, et al.

Serial No.: 10/007,885

Filing Date: November 5, 2001

Title: SILICA-BASED SOLS

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

: Docket: ANO 6119 P1US/3156

: Examiner: Peter Chin

: Group Art Unit: 1731

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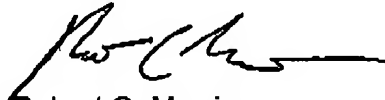
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2. AUTHORIZATION TO ACT IN A REPRESENTATIVE CAPACITY;
3. APPEAL BRIEF;
4. DECLARATION UNDER 37 C.F.R. § 1.132 (2)
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Respectfully submitted,



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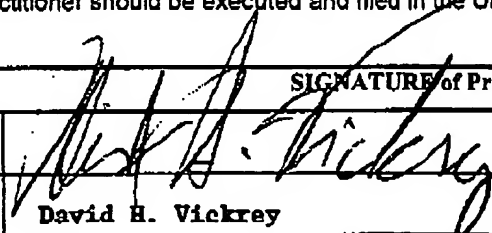
AUTHORIZATION TO ACT IN A REPRESENTATIVE CAPACITYIn re Application of:
PERSSON, M. ET AL.Application No.
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November 5, 2001Title:
Silica-Based SolsAttorney Docket No.
ANO 6119 PLUS/3156Art Unit:
1731

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SIGNATURE of Practitioner of Record

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Serial No. 10/007,885

PATENT**RECEIVED
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BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES**In re Application of:
PERSSON, et al.

Serial No.: 10/007,885

Filing Date: November 5, 2001

Title: SILICA-BASED SOLS

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

: Docket: ANO 6119 P1US

: Examiner: Peter Chin

: Group Art Unit: 1731

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Commissioner for Patentson July 24, 2006Lynn Brush
Lynn Brush**BRIEF ON APPEAL**

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Lynn Brush

This Appeal Brief is submitted to appeal the decision dated August 26, 2005 of the Examiner finally rejecting all of the pending claims. A Notice of Appeal was filed on February 23, 2006. Accordingly, this Brief is being submitted along with a Petition for a three month extension of time.

I. INTRODUCTION

Pursuant to the provisions of 35 U.S.C. §134 and 37 C.F.R. § 41.37, this paper is submitted as a brief setting forth the authorities and arguments upon which Appellants rely in support of the Appeal from the Final Rejection of claims 1 and 22-40 entered in the above-identified patent application on August 26, 2005.

II. REAL PARTY IN INTEREST

The real party in interest in the present case is Akzo Nobel nv, Arnhem, The Netherlands.

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III. RELATED APPEALS AND INTERFERENCES

There are no related appeals or interferences pending or anticipated involving the present application.

IV. STATUS OF THE CLAIMS

The present application was filed in the United States Patent and Trademark Office on November 5, 2001, as a continuation application of PCT/SE00/00821, with original claims 1-40, along with a preliminary amendment which canceled claims 2-21. Claims 1 and 22-40 stand finally rejected. Accordingly, claims 1 and 22-40 are pending herein on Appeal, and are reproduced in the Claims Appendix to this Brief.

V. STATUS OF THE AMENDMENTS

No amendments were filed subsequent to the Office Action dated August 26, 2005, which finally rejected all of the pending claims.

VI. SUMMARY OF THE CLAIMED SUBJECT MATTER

In the paper making art, drainage and retention aids are conventionally introduced into paper stock in order to facilitate drainage and to increase adsorption of fine particles onto the cellulose fibers so that they are retained with the fibers. Silica-based particles are commonly used as drainage and retention aids in combination with charged organic polymers. The silica-based particles are normally supplied in the form of aqueous colloidal dispersions (sols). Commercial sols usually have a silica content of 7-15 wt% and contain particles having a specific area of at least 300 m²/g. Sols of particles having higher specific surface areas are usually more dilute to improve storage stability and avoid gel formation.

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The present inventors have discovered silica-based sols exhibiting improved high surface area stability, i.e., improved capability to maintain high specific surface area and storage at a relatively high silica concentration. The sols according to the invention also exhibit improved drainage and retention when used in conjunction with conventional charged organic polymers, making it possible to increase the speed of the paper machine and to use lower levels of additives in the paper stock compared to other commercially available sols. (See Example 6, Table 2).

Independent claim 1 relates to an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 10 to 45%; a viscosity within the range of from 5 to 40 cP; and a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 10:1 to 40:1; and wherein the silica-based particles have a specific surface area within the range of from 550 to 725 m^2/g . (See specification at page 2, line 24 to page 3, line 20)

Claims 22-28 all further define the aqueous sol according to claim 1. Claim 22 more narrowly defines the S-value within the range of from 20 to 40% (page 2, lines 27-28). Claim 23 defines the molar ratio of SiO_2 to M_2O within the range of from 15:1 to 30:1 (page 2, line 34). Claim 24 provides the sol has a pH of at least 10.6 (page 2, line 35). Claim 25 more narrowly defines the viscosity within the range of from 7 to 25 cP (page 3, line 9). Claim 26 provides the sol has a molar ratio of Al_2O_3 to SiO_2 within the range of from 1:4 to 1:1500 (page 4, lines 4-6). Claim 27 provides the sol has a molar ratio of B, where B is boron, to SiO_2 within the range of from 1:4 to 1:1500 (page 4, lines 6-8). Finally, claim 28 provides the sol has a molar ratio of Al to B, where B is boron, within the range of from 100:1 to 1:100 (page 4, line 8-10).

Independent claim 29 relates to an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 10 to 45%; a viscosity within the range of from 5 to 40 cP; and a silica content of at least 10% by weight; and wherein

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the silica-based particles have a specific surface area within the range of from 550 to 725 m²/g. (Page 2, line 24 to page 3, line 20)

Claims 30-34 all further define the aqueous sol according to claim 29. Claim 30 more narrowly defines the S-value within the range of from 20 to 40% (page 2, line 27-28). Claim 31 provides that the sol has a pH of at least 10.6 (page 2, line 35). Claim 32 defines that the sol has a silica content within the range of from 12 to 20% by weight (page 3, line 3). Claim 33 more narrowly defines the viscosity within the range of from 7 to 25 cP (page 3, line 9). Claim 34 provides the sol has a molar ratio of SiO₂ to M₂O, where M is alkali metal or ammonium, within the range of from 10:1 to 40:1 (page 2, line 32-33).

Independent claim 35 relates to an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 10 to 45%; a viscosity within the range of from 7 to 25 cP; a silica content of at least 10% by weight; a molar ratio of SiO₂ to M₂O, where M is alkali metal or ammonium, within the range of from 10:1 to 40:1; and a pH of at least 10.6. (Page 2, line 24 to page 3, line 12)

Claims 36-38 all further define the aqueous sol according to claim 35. Claim 36 provides the silica-based particles have a specific surface area of at least 300m²/g up to 1050 m²/g (page 3, lines 17-19). Claim 37 defines the specific surface area within the range of from 775 to 1050 m²/g (page 3, line 21). Claim 38 defines the specific surface area within the range of from 550 to 725 m²/g (page 3, line 20).

Finally, independent claim 39 relates to an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 10 to 45%; a viscosity within the range of from 5 to 40 cP; a silica content of at least 10% by weight; a molar ratio of SiO₂ to M₂O, where M being alkali metal or ammonium, within the range of from 10:1 to 40:1; and wherein the sol is modified by an aluminium-containing compound, a boron-containing compound or a mixture thereof. (Page 2, line 24 to page 3, line 12 and page

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4, lines 2-4) Claim 40 depends from claim 39 and provides that the silica-based particles have a specific surface area of at least $300\text{m}^2/\text{g}$ up to $1050\text{m}^2/\text{g}$ (page 3, lines 17-19).

VII. GROUNDS OF REJECTION TO BE REVIEWED ON APPEAL

The issues remaining in the present case which require resolution herein on Appeal are summarized as follows:

Claims 1 and 22-40 stand rejected under 35 U.S.C. § 103(a) over U.S. Patent No. 5,277,764 to Johansson et al. (hereinafter "Johansson"), U.S. Patent No. 5,607,552 to Andersson et al. (hereinafter "Andersson") or U.S. Patent No. 6,486,216 to Keiser et al. (hereinafter "Keiser").

VIII. ARGUMENT

The Rejection of Claims 1 and 22-40 under 35 USC 103(a) Over Johansson, Andersson or Keiser

In rejecting claims 1 and 22-40, the examiner contends that Johansson discloses that prior art colloidal silica or silica sols have an S value of 8-45%, a $\text{SiO}_2:\text{M}_2\text{O}$ ratio of between 20:1 and 75:1, and a specific area of between 10 and $1000\text{m}^2/\text{g}$, which includes sols modified by Al. Appellants disagree, as discussed more fully below, and respectfully submit that nowhere does Johansson disclose a single prior art sol having all of these properties.

The examiner further contends that Andersson and Keiser also disclose sols having the above-listed properties and which encompass the specific area and S-values as claimed. Appellants again disagree, as discussed more fully below.

The examiner correctly acknowledges that the cited references are silent as to viscosity. However, the examiner contends that since viscosity is dependent on such

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factors as particle size and specific surface area, and since the S-values and specific area of the sols disclosed by the cited references are within the claimed range, the prior art obviously encompasses the claimed viscosity. Appellants disagree.

To establish a *prima facie* case of obviousness, three basic criteria must be met. First, there must be some suggestion or motivation, either in the reference itself or in the knowledge generally available to one of ordinary skill in the art, to modify the reference. Second, there must be a reasonable expectation of success. Finally, the prior art reference (or references when combined) must teach or suggest all claim limitations. See MPEP §2142.

The teaching or suggestion to make the claimed combination (or modification) and the reasonable expectation of success must both be found in the prior art, and not based on the applicant's disclosure. *In re Vaeck*, 947 F.2d 488, 20 U.S.P.Q.2d 1438 (Fed. Cir. 1991).

The initial burden is on the examiner to provide some suggestion of the desirability of doing what the inventor has done. "To support the conclusion that the claimed invention is directed to obvious subject matter, the reference must expressly or impliedly suggest the claimed invention or the examiner must present a convincing line of reasoning as to why the artisan would have found the claimed invention to have been obvious in light of the teachings of the references." *Ex parte Clapp*, 227 U.S.P.Q. 972, 973 (Bd. Pat. App. & Inter. 1985).

A. The Claimed Viscosity is Not Inherently Disclosed

In the instant case, although the examiner acknowledges that the cited references are silent as to the claimed viscosity, it is the examiner's contention that the claimed viscosity is inherent in the prior art (i.e., "the prior art obviously encompasses

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the claimed viscosity") based on prior art sols having S-values and specific surface areas within the claimed ranges. Appellants disagree.

If the claimed viscosity is inherently disclosed (or "obviously encompassed") by the cited references, it must be necessarily present and a person of ordinary skill in the art must recognize its presence. *Crown Operations Intern. Ltd. V. Solutia Inc.*, 289 F.3d 1367, ___, 62 U.S.P.Q.2d 1917, 1922-23 (Fed. Cir. 2002) (citing *In re Robertson*, 169 F.3d 743, 745, 49 U.S.P.Q.2d 1949, 1950-51 (Fed. Cir. 1999)) (Emphasis added). Inherency "may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient." *Id.* at 1923. (quoting *In re Oelrich*, 666 F.2d 578, 581, 212 U.S.P.Q. 323, 326 (C.C.P.A. 1981)).

Appellants respectfully submit that the claimed viscosity is not a necessary result from a sol having an S-value and specific surface area falling within the claimed ranges. Appellants' position is supported by two declarations under 37 C.F.R. §1.132, which were previously filed on June 15, 2005, in response to the Office Action dated December 15, 2004, and which are attached in the Evidence Appendix to this Brief.

In the Declaration of Dr. Marek Tokarz, co-inventor of the present application, Dr. Tokarz presents experimental data relating to aqueous sols containing silica-based particles and the evaluation of these sols by means of drainage and retention performance in papermaking. The aqueous sols tested encompass both prior art sols and sols according to the claims of the present invention.

Dr. Tokarz concludes that it is not correct to assume that, since the S-values and specific surface area of the prior art sols are within the claimed range, the prior art obviously encompasses the claimed viscosity. The test results presented in his declaration demonstrate, *inter alia*, that prior art sols of silica based particles having S-values and specific surface areas within the claimed range can have viscosities outside of the claimed range. (See Tokarz Dec. at page 7, ¶ 9). Specifically, the sol of example

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1c has an S-value of 32 and a specific surface area of 690 m²/g (within the ranges of claim 1), but has a viscosity of 1.6 cP (outside the range of claim 1). (See Tokarz Dec. at page 2, ¶ 6 and Table 1).

In the Declaration of Dr. Hans E. Johansson Vestin, co-inventor of the Johansson and Andersson patents, Dr. Johansson Vestin elaborates on parameters of aqueous sols containing silica-based particles. Dr. Johansson Vestin concludes that it is not correct to assume that, since the S-values and specific surface area of the prior art sols are within the claimed range, the prior art obviously encompasses the claimed viscosity. Dr. Johansson Vestin further opines that viscosity depends on several parameters of an aqueous silica-based sol, not only S-values and specific surface area. As a consequence, according to Dr. Johansson Vestin, it is certainly possible that the aqueous sol containing silica-based particles according to the present invention can have a viscosity that is different, even to a large extent from the viscosity of prior art sols, even if the S-values and specific surface areas are within the same range.

In commenting on these declarations, the examiner noted that although the viscosity was out of the claimed range for example 1c, it was in the claimed range for examples 2a, 2b, 2c, 2d, 2e, 2f, 4, 5a and 5d. The examiner then concludes, based on the latter examples, that "in consideration of the other factors it is obvious that the viscosity will be within the claimed range." Appellants respectfully disagree.

Examples 2a, 2b, 2c, 2d, 2e, 2f, 4, 5a and 5d are all examples of the claimed invention. That is why they fall within the claimed viscosity range. However, a review of the test results presented by Dr. Tokarz demonstrates that the claimed viscosity is not a necessary result from a sol having parameters that otherwise fall within the claimed ranges. In that regard, in addition to having an S-value and specific surface area falling in the claimed ranges (of claim 1), example 1c also has a molar ratio of SiO₂ to M₂O that falls within the claimed range, yet has a viscosity outside the claimed range. Similarly, regarding claim 35, examples 1f and 6b both have S-values, silica contents, molar ratios

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of SiO_2 to M_2O and pH values within the claimed ranges, but have viscosities outside the claimed range. Therefore, it is respectfully submitted that the claimed viscosity is not necessarily present when the other claimed parameters are present.

Appellants respectfully submit that the examiner is using the wrong legal standard for inherency. In order to make a prima facie case of obviousness in the present case, it is the examiner's burden to establish that the prior art sols, having all the other claim limitations, necessarily include the claimed viscosity and that a person of ordinary skill in the art would recognize its presence. See *Crown Operations*, 62 U.S.P.Q.2d at 1922-23. Appellants submit that the examiner has not met this burden.

The cited prior art discloses sols having wide ranges of parameters, such as S-values, silica contents, molar ratios of SiO_2 to M_2O and specific surface areas. The mere possibility that a prior art sol, selected from the wide range of parameters specified in the cited prior art, could have the claimed viscosity is not enough. Appellants submit that one skilled in the art would not appreciate the presently claimed invention from the teachings of the prior art. This is supported by the declaration of Dr. Johansson Vestin where he states that the cited prior art does not teach, suggest or disclose the presently claimed invention. (See Johansson Vestin Dec. at pages 4-5, ¶'s 10-14).

B. The Cited Prior Art Does Not Teach or Suggest the Claimed Invention

Johansson generally discloses prior art sols having a specific surface area of 50–1000 m^2/g , more preferably about 100-1000 m^2/g and usually about 400-600 m^2/g . (See Col. 3, lines 59-66). Johansson further discloses, as an alternative, a prior art sol having an S-value of 8-45% and silica particles having a specific area of 750-1000 m^2/g , where the particles are surface modified with Al to a degree of 2-25%, and a $\text{SiO}_2:\text{M}_2\text{O}$ ratio of 20:1 to 75:1. (See Col. 3, line 66 to Col. 4, line 38). Johansson also discloses "another type" of sol that has a $\text{SiO}_2:\text{M}_2\text{O}$ ratio of 6:1 to 12:1 and a specific surface area

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of 700 to 1200 m²/g. (See Col. 4, lines 39-46). Nowhere does Johansson disclose a prior art sol having an S-value of 8-45%, a SiO₂:M₂O ratio of between 20:1 and 75:1, and a specific area of between 10 and 1000 m²/g, as alleged by the Examiner. It is respectfully submitted that Johansson teaches and fairly suggests that when the sol has an S-value of 8-45% and a SiO₂:M₂O ratio of between 20:1 and 75:1, the specific area should be 750-1000 m²/g. Thus, Appellants submit that Johansson teaches away from claims 1, 29 and 38. Otherwise, Johansson merely teaches sols having a broad range of specific surface area (or a combination of a SiO₂:M₂O ratio of 6:1-12:1 and a specific surface area of 700 to 1200 m²/g), without any suggestion to provide a sol having the claimed parameters.

Andersson is directed to aqueous suspensions containing both silica sol particles and swollen particles of clays with the weight ratio of sol to clay within the range of 20:1 to 1:10. Andersson generally discloses suitable sols having a specific surface area of 50-1000 m²/g, more preferably about 100-1000 m²/g and usually about 400-600 m²/g. (See Col. 2, lines 13-22). Andersson further discloses particularly suitable sols having an S-value of 8-45% and silica particles having a specific area of 750-1000 m²/g, where the particles are surface modified with Al to a degree of 2-25%. (See Col. 2, lines 22-28). Andersson also generally discloses sols having S-values from 15-40% and specific surface areas within the range of 300-700 m²/g, preferably 400-650 m²/g. (See Col. 2, lines 37-43). Appellants submit that Andersson merely teaches sols generally having an S-value from 15-40% and a specific surface area of 400-650 m²/g, and that it fails to suggest any reason to provide a sol having the claimed parameters.

Keiser discloses silica sols having a specific surface area of 700-1100 m²/g, S-value of 20-50%, a SiO₂:Na₂O ratio of 13:1 to 17:1, a SiO₂ content of 7-18.8 wt% and wherein the silica is not surface treated. Although Keiser teaches sols having broad ranges of parameters, with some overlap of the claimed parameters, nowhere does it teach or suggest the claimed viscosity or to modify the disclosed parameters to arrive at

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the claimed invention. Moreover, Appellants submit that Keiser teaches away from claims 26-28 and 39, which include surface modified silica particles.

It is respectfully submitted that the cited prior art merely discloses silica sols having broad ranges of parameters, as discussed above, without any suggestion to modify their teachings to arrive at the claimed invention. A "determination of obviousness cannot be based on the hindsight combination of components selectively culled from the prior art to fit the parameters of the patented invention." *Crown Operations*, 62 U.S.P.Q.2d at 1922 (*quoting ATD Corp. v. Lydall, Inc.*, 159 F.3d 534, 546, 48 U.S.P.Q.2d 1321, 1329 (Fed. Cir. 1998)). There must be a teaching or suggestion in the prior art, within the nature of the problem to be solved, or within the general knowledge of a person of ordinary skill in the field of the invention, to look to particular sources, to select particular elements, and to combine them as combined by the inventor. *Id.* at 1922.

Appellants respectfully submit that, in order to obtain the respective sols as presently claimed (in independent claims 1, 29, 35 and 39), one would have to pick and choose individual properties from the various teachings in the cited prior art references and then select the claimed viscosity, in the absence of any teachings or suggestions to do so. It is respectfully submitted that the only way to accomplish this, is with the improper use of hindsight.


Moreover, the sols according to the presently claimed invention unexpectedly provide improved water drainage compared to various prior art sols. Test results presented in the declaration of Dr. Tokarz show that sols of examples 2d, 3 and 4 according to the invention resulted in improved drainage compare to the prior art sols of examples 1a, 1b, 1c, 6a and 6b. (See Tokarz Dec. at page 6, Table 6) Additional tests show that sols of examples 5a and 5b according to the invention resulted in improved drainage compared to the prior art sol of example 6a. (See Tokarz Dec. at page 7,

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Table 7). Appellants respectfully submit that these unexpected results support a finding that the claimed invention is not obvious over the prior art.

In view of the arguments presented herein Appellants respectfully submit that claims 1 and 22-40 stand improperly rejected. The Honorable Board is therefore respectfully requested to reverse the Examiner and pass all of the pending claims to issue.

Respectfully submitted,



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IX. CLAIMS APPENDIX

1. An aqueous sol containing silica-based particles, which sol has:
 - (i) an S-value within the range of from 10 to 45%;
 - (ii) a viscosity within the range of from 5 to 40 cP; and
 - (iii) a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 10:1 to 40:1; and
 - (iv) the silica-based particles have a specific surface area within the range of from 550 to 725 m^2/g .
22. The aqueous sol according to claim 1, wherein the S-value is within the range of from 20 to 40%.
23. The aqueous sol according to claim 1, wherein the sol has a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1.
24. The aqueous sol according to claim 1, wherein the sol has pH of at least 10.6.
25. The aqueous sol according to claim 1, wherein the sol has a viscosity within the range of from 7 to 25 cP.
26. The aqueous sol according to claim 1, wherein the sol has a molar ratio of Al_2O_3 to SiO_2 within the range of from 1:4 to 1:1500.
27. The aqueous sol according to claim 1, wherein the sol has a molar ratio of B, where B is boron, to SiO_2 within the range of from 1:4 to 1:1500.
28. The aqueous sol according to claim 1, wherein the sol has a molar ratio of Al to B, where B is boron, within the range of from 100:1 to 1:100.

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29. An aqueous sol containing silica-based particles, which sol has:

- (i) an S-value within the range of from 10 to 45%;
- (ii) a viscosity within the range of from 5 to 40 cP; and
- (iii) a silica content of at least 10% by weight; and
- (iv) the silica-based particles have a specific surface area within the range of from 550 to 725 m²/g.

30. The aqueous sol according to claim 29, wherein the S-value is within the range of from 20 to 40%.

31. The aqueous sol according to claim 29, wherein the sol has a pH of at least 10.6.

32. The aqueous sol according to claim 29, wherein the sol has a silica content within the range of from 12 to 20% by weight.

33. The aqueous sol according to claim 29, wherein the sol has a viscosity within the range of from 7 to 25 cP.

34. The aqueous sol according to claim 29, wherein the sol has a molar ratio of SiO₂ to M₂O, where M is alkali metal or ammonium, within the range of from 10:1 to 40:1.

35. An aqueous sol containing silica-based particles, which sol has:

- (i) an S-value within the range of from 10 to 45%;
- (ii) a viscosity within the range of from 7 to 25 cP;
- (iii) a silica content of at least 10% by weight;
- (iv) a molar ratio of SiO₂ to M₂O, where M is alkali metal or ammonium, within the range of from 10:1 to 40:1; and
- (v) a pH of at least 10.6.

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36. The aqueous sol according to claim 35, wherein the silica-based particles have a specific surface area of at least $300\text{m}^2/\text{g}$ up to $1050\text{ m}^2/\text{g}$.

37. The aqueous sol according to claim 35, wherein the silica-based particles have a specific surface area within the range of from 775 to $1050\text{ m}^2/\text{g}$.

38. The aqueous sol according to claim 35, wherein the silica-based particles have a specific surface area within the range of from 550 to $725\text{ m}^2/\text{g}$.

39. An aqueous sol containing silica-based particles, which sol has:

- (i) an S-value within the range of from 10 to 45%;
- (ii) a viscosity within the range of from 5 to 40 cP;
- (iii) a silica content of at least 10% by weight;
- (iv) a molar ratio of SiO_2 to M_2O , where M being alkali metal or ammonium, within the range of from 10:1 to 40:1; and
- (v) the sol is modified by an aluminium-containing compound, a boron-containing compound or a mixture thereof.

40. The aqueous sol according to claim 39, wherein the silica-based particles have a specific surface area of at least $300\text{m}^2/\text{g}$ up to $1050\text{ m}^2/\text{g}$.

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X. EVIDENCE APPENDIX

1. Declaration of Dr. Marek Tokarz Under 37 C.F.R. §1.132, filed along with Applicants' Response of June 15, 2005, to the Office Action dated December 15, 2004.
2. Declaration of Dr. Hans E. Johansson Vestin Under 37 C.F.R. §1.132, filed along with Applicants' Response of June 15, 2005, to the Office Action dated December 15, 2004.

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XI. RELATED PROCEEDINGS APPENDIX

None.

PATENT
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	:	Docket: ANO 6119/3156
PERSSON, et al.	:	Examiner: Peter Chin
Serial No. 10/007,885	:	Group Art Unit: 1731
Filing Date: November 5, 2001	:	
For: SILICA-BASED SOLS	:	

Assistant Commissioner for Patents
Washington, D.C. 20231

DECLARATION UNDER 37 C.F.R. §1.132

Sir:

I, Marek Tokarz, do hereby declare and say that:

1. I am a citizen of Sweden residing at Håkons Gata 4, SE-442 39 Kungälv, Sweden.
2. I have a degree of M.Sc.Eng. from University Academy of Mining and Metallurgy in Krakow, Poland, and a degree of Ph.D. from University of Utah in Salt Lake City, UT, USA.
3. I am a Senior Specialist employed by Eka Chemicals AB since 1984, and since 1990 I have been involved in the development of silica-based sols for use in papermaking.
4. I am a co-inventor of U.S. Patent Application Serial No. 10/007,885 ("the present application") and I am familiar with the fields of silica-based sols and papermaking.
5. I have also read and understood the specifications of U.S. Patent Nos. 5,277,764 (Johansson et al), 5,607,552 (Andersson et al) and 6,486,216 (Keiser et al) cited in the present application.

6. The following tests, relating to the preparation of aqueous sols containing silica-based particles and evaluation of these sols by means of drainage and retention performance in papermaking, have been conducted by me or under my direct supervision:

Three standard aqueous sols containing silica-based particles were prepared by a prior art process according to the following.

762.7 g sodium water glass with a molar ratio of SiO_2 to Na_2O of 3.3 and SiO_2 content of 27.1 % by weight was diluted with water to 3000 g yielding a silicate solution (I) with a SiO_2 content of 6.9% by weight. 2800 g of this silicate or water glass solution was passed through a column filled with a strong cation exchange resin saturated with hydrogen ions. 2450 g of ion-exchanged water glass or polysilicic acid (II) with an SiO_2 content of 6.5% by weight and a pH of 2.4 was collected from the ion exchanger. 1988 g of the polysilicic acid (II) was fed into a reactor and diluted with 12.3 g water. 173.9 g of the 6.9% silicate solution (I) was then added under vigorous agitation. The resulting solution was then heated at 85°C for 60 minutes and then cooled to 20°C to provide a sol of containing silica-based particles. A similar process was used to produce additional sols using somewhat different settings.

Table 1 shows the parameters of the prior art aqueous sols containing silica-based particles.

Table 1

Sol No.	Specific Surface Area (m ² /g)	S-value [%]	Viscosity [cP]	pH	Molar Ratio $\text{SiO}_2:\text{Na}_2\text{O}$	Content of Silica [% by weight]
1a	530	29	2.2	10.2	40	7.3
1b	500	26	2.7	10.0	63	7.3
1c	690	32	1.8	9.8	35	5.4

Three additional aqueous sols containing silica-based particles were prepared by a prior art process comprising acidification of an aqueous sodium silicate solution to a pH below 4 and subsequent alkalinization to a pH of at least 9:

The aqueous sodium silicate solution, which had a molar ratio SiO_2 to Na_2O of about 3.5, was acidified to a pH below 4 by means of an acid cation exchanger saturated with hydrogen ions to form a polysilicic acid. The polysilicic acid was subjected to alkalization by means of addition of an aqueous sodium silicate solution having a molar ratio SiO_2 to Na_2O of about 3.5. The degree of microgel was influenced by, inter alia, keeping the SiO_2 content during the alkalization in the range of from 4.5 to 7.0% by weight.

Table 2 shows the parameters of the prior art aqueous sols containing silica-based particles.

Table 2

Sol No.	Specific Surface Area [m ² /g]	S-value (%)	Viscosity [cP]	pH	Molar Ratio $\text{SiO}_2/\text{Na}_2\text{O}$	Content of Silica [% by weight]
1d	850	30	3.1	9.0	30	7.8
1e	850	24	5.0	9.0	45	7.4
1f	870	35	2.5	10.6	10	10.0

7. Six aqueous sols containing silica-based particles according to the invention claimed in the present application were prepared from a polysilicic acid similar to the polysilicic acid (II) according to point 6 above produced with the same ion exchange process and with an SiO_2 content of 5.46 % by weight. To 102.0 kg of the polysilicic acid was added 1.46 kg of sodium water glass with a ratio $\text{SiO}_2/\text{Na}_2\text{O}$ of 3.3 under vigorous agitation resulting in a solution with a molar ratio $\text{SiO}_2/\text{Na}_2\text{O}$ of 54.0. This solution was heat treated at 60°C for 2 h 20 min and cooled to 20°C whereupon the product was concentrated to a SiO_2 content of 15.6 % by weight. This intermediate sol product was now divided into six separate samples, a to f. Samples a to c were further alkalisied with NaOH, samples d to f with water glass, to achieve sols with a molar ratio $\text{SiO}_2/\text{Na}_2\text{O}$ between 21.5 and 34.0 and a silica content of about 15.0 % by weight.

Table 3 shows the parameters of the aqueous sols containing silica-based particles according to the invention claimed in the present application:

Table 3

Sol No.	Specific Surface Area [m ² /g]	S-value [%]	Viscosity [cP]	pH	Molar Ratio SiO ₂ :Na ₂ O	Content of Silica [% by weight]
2a	720	31	17	10.7	21.5	about 15
2b	710	30	29	10.3	28.0	about 15
2c	690	29	40	10.0	34.0	about 15
2d	680	31	20	10.7	21.5	about 15
2e	670	29	34	10.3	28.0	about 15
2f	680	29	38	10.0	33.0	about 15

Three additional aqueous sols containing silica-based particles according to the invention claimed in the present application, Sols 3, 4, 5a and 5b, were produced as follows:

A polysilicic acid (II) produced with the above ion exchange process and alkalisied with water glass to a molar ratio SiO₂/Na₂O of 54.0 as under point 6 above was heat treated at 60°C for 1 h. To 58 kg of this product was added 7.25 kg of diluted water glass with a molar ratio SiO₂/Na₂O of 3.3 and silica content 5.5 % by weight. The resulting sol of silica-based particles, Sol 3, was then concentrated.

1000 g polysilicic acid (II) with an SiO₂ content of 5.5 % by weight was mixed with 14.5 g water glass solution with an SiO₂ content of 27.1 % by weight and a molar ratio SiO₂/Na₂O = 3.3 under vigorous agitation resulting in a product with a molar ratio SiO₂/Na₂O of 51 and a silica content of 5.8 % by weight SiO₂, which was heat treated at 60°C for 1.5 h and then concentrated to a silica content of 16.7 % by weight SiO₂. 283 g of the product obtained was mixed with 33.0 g NaOH resulting in a sol of silica-based particles, Sol 4.

The general procedure used for Sol 4 was followed except that the heat treatment was carried out for 1.25 h and concentration was carried out to higher silica contents. Two sols of silica-based particles were prepared; Sol 5a and Sol 5b.

Table 4 shows the parameters of Sols 3, 4, 5a and 5b according to the invention claimed in the present application:

Table 4

Sol No.	Specific Surface Area [m ² /g]	S-value [%]	Viscosity [cP]	pH	Molar Ratio SiO ₂ :Na ₂ O	Content of Silica [% by weight]
3	760	34	9.0	10.7	24	15.2
4	720	32	14.2	10.6	21	15.2
5a	700	36	18	10.7	18	18
5b	700	37	31	10.7	18.3	20

7. Drainage performance was evaluated by means of a Dynamic Drainage Analyser (DDA), available from Akribi, Sweden, which measures the time for draining a set volume of stock through a wire when removing a plug and applying vacuum to that side of the wire opposite to the side on which the stock is present.

The stock used was based on a blend of 60 % by weight bleached birch sulphate and 40 % by weight bleached pine sulphate to which was added 30 % by weight ground calcium carbonate as a filler. Stock volume was 800 ml, consistency 0.25 % by weight and pH about 8.0. Conductivity of the stock was adjusted to 0.47 mS/cm by addition of sodium sulphate.

In the tests, silica-based sols were used in conjunction with a cationic polymer, Raisamyl 142, which is a conventional medium-high cationised starch having a degree of substitution of 0.042, which was added to the stock in an amount of 12 kg/tonne, calculated as dry starch on dry stock system. Silica-based sols described above were tested and compared with standard sols 6a and 6b. Table 5 shows the parameters of these prior art aqueous sols containing silica-based particles.

Table 5

Sol No.	Specific Surface Area [m ² /g]	S-value [%]	Viscosity [cP]	pH	Molar Ratio SiO ₂ :Na ₂ O	Content of Silica [% by weight]
6a	500	45	3.0	10.2	40	15.0
6b	880	36	2.5	11	10	10.0

The silica-based sols were added in an amount of 0.5 kg/ton, calculated as SiO_2 and based on dry stock system. The stock was stirred in a baffled jar at a speed of 1500 rpm throughout the test and chemical additions were conducted as follows: i) adding cationic starch to the stock following by stirring for 30 seconds, ii) adding silica-based sol to the stock followed by stirring for 15 seconds, iii) draining the stock while automatically recording the drainage time. Drainage times for the different silica-based sols are shown in Table 6:

Table 6

Silica-based sol	Drainage time [sec]
Sol 1a (ref.)	12.0
Sol 1b (ref.)	11.1
Sol 1c (ref.)	12.0
Sol 2d	9.7
Sol 3	9.5
Sol 4	9.4
Sol 6a (ref.)	12.0
Sol 6b (ref.)	9.8

Table 6 shows that the aqueous sols containing silica-based particles according to the invention claimed in the present application provided improved drainage times.

8. Drainage performance was evaluated according to the general procedure described under point 7 above except that the stock had a consistency of 0.3% and pH about 8.5. Retention performance was evaluated by means of a nephelometer by measuring the turbidity of the filtrate, the white water, obtained by draining the stock.

Silica-based sols according to invention claimed in the present application were tested against Sol 6a used for comparison. Table 7 shows the drainage time obtained at various dosages (kg/ton) of silica-based particles, calculated as SiO_2 and based on dry stock system. The addition of only cationic starch (12 kg/tonne, calculated as dry starch on dry stock system) resulted in a drainage time of 15.8 sec.

7

Table 7

Silica-based sol	Drainage time (sec) / Turbidity (NTU) at SiO ₂ dosage of				
	0.5 kg/t	1.0 kg/t	1.5 kg/t	2.0 kg/t	3.0 kg/t
Sol 6a (ref.)	11.1/ -	8.8/59	7.9/58	7.1/54	6.8/60
Sol 5a	9.0/ -	7.1/52	6.3/50	5.2/52	5.7/53
Sol 5b	8.9/ -	6.9/ -	6.3/ -	5.7/ -	6.0/ -

Table 7 shows that the aqueous sols containing silica-based particles according to the invention claimed in the present application provided improved drainage times.

9. In an Office Action mailed December 15, 2004 in respect of the present application, it is stated "since viscosity is dependent on such factors as particle size and surface area, it is obvious that since the S-values and specific surface area of the above prior art sols are within the claimed range, the prior art obviously encompasses the claimed viscosity".

In my professional opinion, it is correct that viscosity is dependent on such factors as particle size and surface area. However, I conclude that it is not correct that since the S-values and specific surface area of the above prior art sols are within the claimed range, the prior art obviously encompasses the claimed viscosity. The above tests show prior art sols of silica-based particles having S-values and specific surface areas within the claimed range but having viscosities outside the claimed range.

10. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date:

13-06-2005

Name:

Marek Tokarz

Marek Tokarz

PATENT
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	:	Docket: ANO 6119/3156
PERSSON, et al.	:	Examiner: Peter Chin
Serial No. 10/007,885	:	Group Art Unit: 1731
Filing Date: November 5, 2001	:	
For: SILICA-BASED SOLS	:	

Assistant Commissioner for Patents
Washington, D.C. 20231

DECLARATION UNDER 37 C.F.R. §1.132

Sir:

I, Hans E. Johansson Vestin, do hereby declare and say that:

1. I am a citizen of Sweden residing at Madängsgatan 5, SE-442 33 Kungälv, Sweden. Previously, my name was Hans E. Johansson but in 1997, I added my wife's family name Vestin to my family name Johansson.
2. I have a degree of Doctor of Philosophy, chemistry, from the Chalmers University of Technology in Gothenburg, Sweden.
3. I am a Senior Research Scientist employed by Eka Chemicals AB, formerly Eka Nobel AB and Eka AB, in Sweden since 1973, and since 1979 I have been continually involved in research and development concerning drainage and retention aids, including silica-based sols, for use in papermaking.
4. I have read and understood the specification of U.S. Patent Application Serial No. 10/007,885 ("the present application") relating to silica-based sols.

5. I have also read and understood the specifications of U.S. Patent Nos. 5,277,764 (Johansson et al; "the '764 patent"), 5,607,552 (Andersson et al; "the '552 patent") and 6,486,216 ("Keiser et al") cited in the present application. I am a co-inventor of the '764 and '552 patents.

6. The '764 patent discloses a process for the production of cellulose fibre containing products in sheet or web form from a suspension of cellulose containing fibres, and optional fillers, which comprises forming and dewatering of the suspension on a wire and drying whereby anionic inorganic particles and a cationic carbohydrate polymer are added to the suspension.

The anionic inorganic particles can be silica-based particles and the '764 generally discloses sols of colloidal silica-based particles. However, the '764 patent is silent about the viscosities of the silica-based sols.

7. The '552 patent discloses aqueous stable suspension of colloidal particles, wherein the particles are both silica-based anionic particles and hydrated particles of clays of smectite type which are expandable in water, whereby the weight ratio of silica based particles to clay particles is within the range of from 20:1 to 1:10 and the dry content of the suspension is within the range of from 5 to 40 per cent by weight.

The '552 patent further generally discloses sols of colloidal silica-based particles. However, the '552 patent is silent about the viscosities of the silica-based sols.

8. Keiser et al discloses stable aquasols comprising colloidal silica having a surface area of from about 700 m²/g to about 1100 m²/g, and an S-value of from about 20 to about 50; wherein said colloidal silica is not surface treated; wherein the molar ratio of SiO₂:Na₂O in said colloidal silica is greater than about 13.0:1 and is less than about 17.0:1; and wherein said aquasols have a percent by weight SiO₂ solids level of from about 7.00 percent to about 16.80 percent.

However, Keiser et al is silent about, inter alia, the viscosities of the aquasols.

9. Aqueous sols containing silica-based particles, also referred to herein as aqueous silica-based sols, can be characterized by means of many different parameters. Examples of such parameters include S-value; content of silica; contents of various elements like alkali metals and aluminium; viscosity; density; pH; whether, how and to what extent the sols or silica-based particles have been modified by means of various elements or compounds; particle size and specific surface area of the silica-based particles; etc.

Some of the parameters of aqueous sols containing silica-based particles are independently changeable whereas others are not. For two parameters that are independently changeable, a change in one parameter will not affect the other. For two parameters that are not independently changeable, a change in one parameter will result in a change in the other. However, the resulting change need not be proportional. In addition, if one or even two parameters of an aqueous sol containing silica-based particles are known, it is most often impossible to predict the value of other parameters of the aqueous sol. Furthermore, some parameters of an aqueous sol containing silica-based particles may be independently changeable within certain ranges, even if these parameters are not completely independently changeable.

In an Office Action mailed December 15, 2004 in respect of the present application, it is stated "since viscosity is dependent on such factors as particle size and surface area, it is obvious that since the S-values and specific surface area of the above prior art sols are within the claimed range, the prior art obviously encompasses the claimed viscosity".

It is correct that viscosity is dependent on such factors as particle size and surface area. However, in my professional opinion, it is not correct that since the S-values and specific surface area of the above prior art sols are within the claimed range, the prior art obviously encompasses the claimed viscosity. My opinion is based on the fact that viscosity depends on several factors, or parameters of an aqueous silica-based sol, not only S-values and specific surface area. As a consequence, it is certainly possible that the aqueous sol containing silica-based particles according to the present application has a viscosity which is different, even to a large extent, from the viscosity of prior art sols, even if the S-values and specific surface areas are within the same range.

10. In my professional opinion, the '764 and '552 patents and the Keiser et al references do not teach, suggest or disclose, either explicitly or implicitly, an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 10 to 45%; a viscosity within the range of from 5 to 40 cP; a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 10:1 to 40:1; and the silica-based particles have a specific surface area within the range of from 550 to 725 m^2/g .

11. In my professional opinion, the '764 and '552 patents and the Keiser et al references do not teach, suggest or disclose, either explicitly or implicitly, an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 10 to 45%; a viscosity within the range of from 5 to 40 cP; and a silica content of at least 10% by weight; and the silica-based particles have a specific surface area within the range of from 550 to 725 m^2/g .

12. In my professional opinion, the '764 and '552 patents and the Keiser et al references do not teach, suggest or disclose, either explicitly or implicitly, an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 10 to 45%; a viscosity within the range of from 7 to 25 cP; a silica content of at least 10% by weight; a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 10:1 to 40:1; and a pH of at least 10.6.

13. In my professional opinion, the '764 and '552 patents and the Keiser et al references do not teach, suggest or disclose, either explicitly or implicitly, an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 10 to 45%; a viscosity within the range of from 5 to 40 cP; a silica content of at least 10% by weight; and a molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 10:1 to 40:1; and the sol is modified by an aluminium-containing compound, a boron-containing compound or a mixture thereof.

14. In my professional opinion, the '764 and '552 patents and the Keiser et al references do not teach, suggest or disclose, either explicitly or implicitly, an aqueous sol containing silica-based particles, which sol has an S-value within the range of from 20 to 40%; a viscosity within the range of from 7 to 25 cP; pH of at least 10.6; molar ratio of SiO_2 to M_2O , where M is alkali metal or ammonium, within the range of from 15:1 to 30:1; silica content of at least 10% by weight; and colloidal anionic silica-based particles with a specific surface area within the range of from 550 to 1050 m^2/g .

15. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: June 14, 2005

Name: Hans E. Johansson Vestin
Hans E. Johansson Vestin